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Evaluation of recovered vegetable oil as a biodiesel feedstock

A. Fröhlich† and B. Rice
Teagasc, Oak Park Research Centre, Carlow

About 7,000 t of recovered vegetable oil (RVO) are collected in Ireland each year. The use of this material in animal feed has been banned under recent EU legislation because of food safety and animal health concerns. In order to assess its potential as a biodiesel (fatty acid methyl ester) feedstock, twenty-two 350-kg batches, with a wide range of acid values, were esterified. Ester properties were largely within specification, though variable low-temperature properties sometimes caused the material to be unsuitable for winter use. This problem could be overcome by blending with mineral diesel, but the addition of two pour-point depressants had little effect. Total glycerol concentrations in some batches were high; it could be improved by small changes in the process. Ester yields were low, due mainly to dissolution of ester in the glycerol, but also to saponification of the triglycerides. Tests of the biodiesel in vehicles showed no adverse effects on engine or lubricating oil. Specific fuel consumption was slightly higher than with mineral diesel, which would be expected with biodiesel from any feedstock. The results indicate that it is possible to produce biodiesel of acceptable quality from RVO. Where ester low-temperature properties are inadequate, the problem can be overcome by blending with mineral diesel.

Keywords: Biodiesel: recovered vegetable oil

Introduction

Interest in the use of recovered vegetable oil (RVO) as a biodiesel feedstock has been stimulated by several recent developments. Firstly, concerns about food safety and animal health have led to EU legislation which bans the use of RVO in animal feed rations. Secondly, restrictions on the disposal of organic materials in land-fills is forcing more vegetable-oil users to find other outlets for their waste oil. Finally, there are pressures to increase energy production from renewable sources, and the European Commission has introduced directives proposing to set

†Corresponding author: afrölich@oakpark.teagasc.ie

mandatory targets for transport biofuel production (Commission of the European Communities, 2001). While rapeseed oil grown on set-aside land is widely accepted as a biodiesel feedstock, it is expensive and limited in supply. Alternative, lower-cost feedstocks are desirable, provided the quality of the biodiesel produced from these sources reaches an acceptable standard. The quality requirements of fatty acid methyl esters for use as engine fuel have been defined in national standards such as the Austrian standard (ÖNORM 1191, 1997) and in a draft EU Standard (prEN 14214) which is nearing the end of the ratification process (European Committee for Standardisation, 2001).

Irish RVO collectors estimate that about 7000 t of RVO are collected annually at present. They also estimate that this could increase to 10,000 t if collection systems were improved and restrictions on dumping were tightened. Biodiesel of mainly satisfactory quality has been produced in Austria from 40 RVO samples; in vehicle trials, engine performance and emissions on RVO ester were found to be similar to rape methyl ester (Mittelbach, 1996; Sams, Tieber and Mittelbach, 1996). Subsequently about 15,000 t of biodiesel of acceptable quality was produced in Austria from mainly domestic RVO collected in 175 districts (Mittelbach, 2002*)*. However, RVO quality may be expected to vary from country to country, depending on the oil source, cooking practices and oil collection systems. Local evaluation of RVO properties is therefore required. The objectives of the present work were: (i) to determine the properties relevant to biodiesel production of the RVO collected in Ireland; (ii) to determine the properties of the biodiesel produced from this material; (iii) to define process parameters for the achievement of high yields of good quality biodiesel from this material; (iv) to examine the role of additives and blending to obtain adequate low-temperature properties; (v) to examine the performance of vehicles running on fuels made up mainly of RVO ester.

Materials and Methods

Oil procurement and preparation

Oil samples for the trials were taken from nine 20-t batches of RVO that had been collected from caterers in small (mainly 25 to 50 l) containers. After steam injection and coarse screening, settled water was drained off and RVO samples taken.

Prior to transesterification, the RVO was heated to 120 °C to bring the water concentration below 5 g/kg. Fatty-acid-free RVO was prepared for laboratory use by the addition of sufficient potassium hydroxide in 700 g/l aqueous solution, at 30 to 35 °C, to neutralise the free fatty acids. The precipitated solids were allowed to settle overnight and the clear oil was decanted. The free fatty acid (FFA) concentration of RVO samples was determined according to ISO 660 (1996).

Transesterification

Transesterification ratios of RVO, methanol and potassium hydroxide were obtained from the literature (Freedman, Pryde and Mounts, 1984a; Maurer, 1991), and reaction conditions and purification of the methyl ester were worked out in our laboratory. The following methods were found to give acceptable quality of methyl ester in good yields.

Laboratory transesterification was carried out in a 250-ml conical flask equipped with a magnetic stirrer. Either 1.8 g potassium hydroxide (low potassium hydroxide) dissolved in 33.5 ml methanol (Freedman *et al.,* 1984a) or 2.5 g potassium hydroxide (high potassium hydroxide) dissolved in 24 ml methanol (Maurer, 1991) was added to

120 g of vigorously stirred RVO at 35 to 45 °C. Stirring was continued for 1 h at the same temperature, the mixture was then transferred to a separatory funnel and the glycerol was allowed to separate for a minimum of 3 h. After draining off the glycerol, the methyl ester was transferred into a clean separatory funnel. It was washed gently with one volume of water, and after the layers separated completely (*ca.* 1 h) residual water and methanol were evaporated at 110 °C.

Pilot-plant-scale transesterification was carried out similarly, except that 350 kg of RVO was used with the corresponding amounts of methanol and potassium hydroxide, stirring was carried out by recirculation, and both the glycerol and water layers were allowed to separate overnight. Residual water was removed by heating, and in some batches methanol was removed by distillation after separation of the glycerol phase. Before transesterification, the RVO was heated to 40 °C. Over 7 t of RVO was transesterified in twenty-two 250- to 350-kg batches.

Analytical methods

FFA concentration of RVO samples was determined according to ISO 660 (1996). Fatty acid composition of RVO samples was determined by gas chromatography using a 3% SP2310/2% SP2300 on Chromosorb W AW glass column, 1800 mm × 3 mm (Supelco, Sigma-Aldrich Ireland Ltd.), and flame ionisation detector. The initial column temperature, 190 °C, was held for 6 min, and then increased to 220 °C at 1 °C/min. Injector temperature was 225 °C.

The properties of the methyl esters produced were determined by methods specified in ÖNORM C1191 (1997) or by equivalent British Standard methods. Acid value, viscosity, Conradson carbon residue (CCR), water concentration, cold filter plug point (CFPP) and iodine value were determined by methods given in ISO 660 (1996), ISO 3104 (1976), ISO 10370 (1993), ISO 12937 (2000), EN 116 (European Committee for Standardisation, 2001) and DIN 53 241 (Deutsches Institut für Normung, 1991), respectively. Methods given in BS 2000 (Parts 15, 219 and 223) were used to determine pour point, cloud point and the ash concentration, free and total glycerols were determined enzymatically (Bailer and de Hueber, 1994). Methanol was determined by gas chromatography using a Porapak Q column (900 mm × 3 mm at 90 °C, injector at 110 °C, 25 ml/min nitrogen flow) with a flame ionisation detector, and density was determined with a 50 ml calibrated density bottle.

To determine the fatty acid concentration of the glycerol phase, 10 g of this material was diluted with 30 ml of water and acidified (pH<2) with sulphuric acid (3.0 mol/l). The acidified mixture was extracted twice with 20 ml hexane and once with 20 ml diethyl ether. After evaporation of the solvent on a water-bath, the residue was dissolved in 30 ml ethanol. Fatty acid concentrations were determined by titration with potassium hydroxide (1.0 mol/l) to the phenolphthalein end point, and the methyl ester concentration semi-quanlitatively by thin layer chromatography (Christopherson and Glass, 1968). The weight of fatty acid in the glycerol phase was calculated by multiplying the obtained fatty acid concentration with the molar weight of the fatty acid (i.e. weight equivalent to 1 mol of potassium hydroxide). The molar weights of the fatty acid and of the methyl ester were obtained from the saponification equivalent (Shriner, Fuson and Curtin, 1964), which was determined by saponifying methyl ester (5 g) according to a standard method (AOAC, 1984).

Lubricating oils were screened for the presence of methyl esters by thin layer chromatography (Christopherson and Glass, 1968; Freedman, Pryde and Kwolek, 1984b) using 10% solution in hexane, and if detected they were determined quantitatively as follows. Lubricating oil (5 g) was saponified according to a standard method used for oils (AOAC, 1984), and the concentration of methyl ester was determined from the amount of potassium hydroxide neutralised during saponification (Shriner, Fuson and Curtin, 1964). The weight of the methyl ester in the oil was calculated by multiplying the concentration of methyl ester by its molar weight (see preceding paragraph).

Pour-point depressants

Pour-point depressants were obtained from Lubrizol Ltd. (Merseyside, UK) and Elf Aquitaine (Tour Elf, La Defence, Paris, France). Weighed amounts were stirred into 100 g of methyl ester, and diesel/methyl ester blends were made up by mixing the required amounts of the two components.

Vehicle trials

Two light transport vehicles were operated for one oil-change interval on fuels containing mainly RVO methyl ester. The vehicles were: (i) Peugeot 360 Xad saloon with 1.905 l normally-aspirated indirect injection engine, 34,000 km driven before test; (ii) Isuzu Trooper UBS55 4-wheel drive with direct-injection turbo-charged 2.771 l engine, 231,000 km driven before test.

Vehicle monitoring included engine lubricating oil condition (methyl ester content, viscosity and wear metals), fuel economy and practical observations of vehicle behaviour. Fuel economy was measured for each vehicle during routine use throughout an oil-change interval by logging fuel added and distance travelled. Two of the fuel blends were compared with mineral diesel in separate trials over two similar rural road circuits of 58 and 101 km, respectively, in the Peugeot 360 Xad. Each trial consisted of three runs with each fuel in a fully randomised sequence. After each run the fuel needed to bring the tank contents back to a predetermined level was measured. The lubricating oil viscosities and analyses for wear metal were carried out in the laboratories of Iarnrod Eireann, Inchicore, Dublin 8. Wear metal concentrations (mg/kg) in the lubricating oil were considered to be excessive if above the following concentrations: 150 for Na, Fe and Cu, 25 for Si, 20 for Cr and Al.

Results

Raw material properties

The fatty acid composition of the nine different RVOs used in the present work varied from batch to batch: based on the fatty acid composition it appeared that in most cases rapeseed oil was the predominant component (Table 1). Other RVO properties also varied considerably, acid values ranged from 1.9 to 7.4 (Table 2), and water concentrations from 10 to 50 g/kg.

Table 1. Fatty acid composition of recovered vegetable oil (RVO) samples and rapeseed oil

Fatty acid	RVO	Rapeseed
$C_{16:0}$	4.7 to 14.5	6.1
$C_{16:1}$	0 to 2.4	
$C_{18:0}$	1.4 to 5.9	2.3
$C_{18:1}$	52.9 to 65.8	56.0
$C_{18:2}$	15.4 to 20.7	24.2
$C_{18:3}$	4.1 to 8.8	6.5
$C_{20:0}$	0.6 to 1.0	0.3
$C_{20:1}$	0.4 to 2.3	0.3

Methyl ester properties

The properties of the methyl esters obtained from RVO were compared to the

limits in ÖNORM C 1191 (1997), the first national standard for fatty acid methyl esters as engine fuels. Similar conclusions would have emerged if the draft EN standard, prEN14214, had been used (European Committee for Standardisation, 2001).

In spite of the varying quality of the RVO, the properties of the methyl esters were within specifications (Table 2) with the exception of CCR. To achieve these results, water had to be removed from the starting material, and additional potassium hydroxide was used for the transesterification of RVO with an acid value over 6.0. When the water concentration was not sufficiently reduced or additional potassium hydroxide was not used, total glycerol concentration was above the specified value. It was shown later in the laboratory that the total glycerol concentration in each batch with acid value above 6.0 could be reduced to below the specified limit by the addition of sufficient potassium hydroxide to neutralise the free fatty acids.

The high CCRs obtained in the present work were probably due to high polymer concentrations arising from the long heating period of the Irish RVOs. Mittelbach and Enzelsberger (1999) showed that the polymer concentration in rapeseed oil increased with heating time, which in turn increased the polymer concentration, viscosity and the CCR of the methyl ester derived from this oil. The authors also showed that methyl ester with viscosity above 4.5 mm^2/s, derived from heated rapeseed oil, will have sufficiently high polymer concentration to increase the CCR value above the specified maximum of 0.05. Considering that the RVO used here is derived mainly from rapeseed oil (Table 1) and the viscosities of the RVO methyl esters are mostly above 4.5 mm^2/s (Table 4), polymer concentrations in these esters will probably be high enough to increase the CCR values above 0.05.

The results suggest that the viscosity of occasional samples would be above the ÖNORM C1191 (1997) limit of 5.0 mm^2/s at 40 °C. However, the average viscosity in bulk lots should be well within the ÖNORM C1191 (1997) limit. The iodine

Table 2. Recovered vegetable oil (RVO) and RVO methyl ester properties

	Range	Average (n=22)	ÖNORM C 1191 (in SI units)
RVO properties:			
Acid value	1.9 to 7.4	5.3	
Yield of crude methyl ester (g/kg RVO)	630 to 870	783	
Yield of purified methyl ester (g/kg RVO)	590 to 860	730	
RVO methyl ester properties:			
Acid value	0.02 to 0.73	0.28	≤0.8
Iodine no.	99 to 118	105	≤115
Free glycerol (g/kg)	0.03 to 0.2	0.075	≤0.2
Total glycerol (g/kg)	1.0 to 5.7	2.4	2.4
Methanol content (g/kg)	0 to 1.5	0.4	£2.0
Water content (g/kg)	0.7 to 1.9	1.1	
Density (g/cm^3)	0.877 to 0.887	0.883	0.87–0.89
Kinematic viscosity, 40 °C (mm^2/s)	4.5 to 5.1	4.9	3.5–5.0
Cold filter plug point (°C)	0 to –14	–5.3	≤0 summer ≤–15 winter
Ash (g/kg)	0 to 0.19	0.07	0.2
Conradson carbon residue (total sample)	0.044 to 0.104	0.065	0.05

values (99 to 117) were similar to those of rapeseed oil methyl ester. Average cold filter plug point (CFPP) was –5.3 °C, but varied over a wide range (0 °C to –14 °C). One of the factors identified, as contributing to this variation, was the fatty acid composition of the ester. Higher proportions of the high melting-point saturated fatty acids led to higher CFPP values (Figure 1).

Ester yields and losses: While it was possible to prepare methyl ester of acceptable quality from RVO with simple methods of transesterification, yields were low (Table 2). This was partly due to excessive losses during purification (caused by limitations in the reactor design), but even the yields of crude methyl ester were low (630 to 870 g/kg RVO).

An attempt was made to establish if the low yields were due to the reactivity of the RVO or to impurities in the material. Fatty acids were removed from very poor quality RVO (acid value 8.0) in the laboratory and the fatty-acid-free product was transesterified using two concentrations of potassium hydroxide. The results showed that it was possible to obtain high yields of methyl ester from fatty-acid-free RVO, though somewhat lower than from refined rapeseed oil (Table 3). Transesterification of crude RVO gave much lower yields by both methods.

Yield losses during esterification can be caused by saponification of the triglycerides to salts of fatty acids, and dissolution of the methyl ester in the glycerol phase. Determination of fatty acids and methyl esters in the glycerol phase indicated that yield losses were attributed to both factors. Fatty acid concentration of the glycerol phase was higher with each esterification than expected from the fatty acid concentration of the starting material, hence some triglyceride must have been saponified (Table 4). The methyl ester concentration of the glycerol phase increased with increasing fatty acid concentration of the glycerol phase, which indicated that methyl ester was dissolved in the glycerol by the potassium salts of fatty acids.

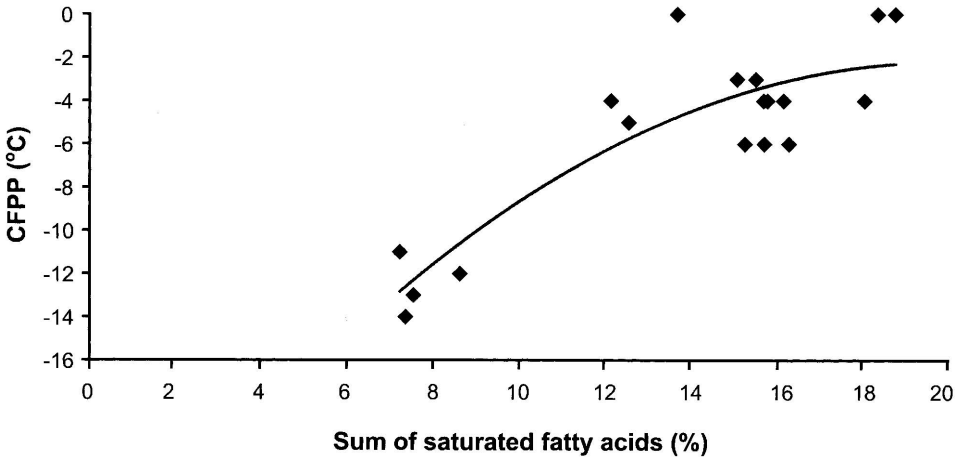

Figure 1: Effect of saturated fatty acid content on cold filter plug point (CFPP) of methyl esters produced from different batches of recovered vegetable oil (RVO).

Table 3. Yields of methyl ester (g/kg) from recovered vegetable oil (RVO) and fatty acid free RVO

Raw material	Transesterification method	
	Low potassium hydroxide	High potassium hydroxide
Fatty acid free rape oil	985[1]±4[2]	926±8
Fatty acid free RVO	973±12	913±5
RVO	839±9	777±7

[1]Average of five esterifications.
[2] 95% confidence interval.

Improvement of low-temperature properties

The CFPP of the methyl ester produced should be adequate for the climate in which it is to be used. Table 5 shows the average ambient temperature distribution for an inland region of Ireland. On average, the occurrence of temperatures below –10 °C is less than 1 h per year. From this it would appear that a CFPP of –10 °C would be very safe, and that a value of –8 °C (exceeded only 4.8 h per year) should very rarely cause problems. For summer use, a CFPP limit of 0 °C should be adequate.

Pour-point depressant additives were effective in lowering the pour points and CFPPs of two RVO samples, but they had only a slight effect on the cloud points (Table 6). However, the RVOs used here were derived mainly from rapeseed oil (Table 1), but tallow occasionally dissolves in vegetable oil during cooking. It was reported (Rice, Fröhlich and Leonard, 1997) that pour-point depressants did not lower the CFPPs of tallow methyl esters, and mixtures of tallow and vegetable oil methyl esters could be similarly affected.

The low-temperature properties of blends with varying proportions of methyl ester and mineral diesel oil suggest that a blend with roughly equal amounts of RVO ester and mineral diesel would have adequate low-temperature properties for Irish winter conditions (Tables 5 and 7). Again cloud point was unaffected, but CFPP and pour point were lowered in proportion to the amount of mineral diesel in the blend (Table 7).

Vehicle tests

For these trials, ester blends containing mainly RVO methyl ester were chosen to give CFPP values from –3 to –8 °C, while keeping the iodine number below 115 (Table 8). All blends were used for at least one oil-change interval. Fuel No. 3 allowed for substantial use of tallow and

Table 4. Effect of free fatty acid (FFA) concentration (g/kg) in recovered vegetable oil (RVO) and esterification method on yield (g/kg) of methyl ester

Raw material	Transesterification method	FFA concentration in		Methyl ester yield[1]
		RVO	Glycerol	
Fatty acid free RVO	Low KOH	0.3	22.2[1]±4.3[2]	973[1]
	High KOH	0.3	50.6±6.0	913
RVO	Low KOH	39.1	67.7±2.0	839
	High KOH	39.1	94.4±1.3	777

[1]Average of five esterifications.
[2]95% confidence interval.

Table 5. Occurrence of low temperatures at an inland Irish meteorological station (Kilkenny, mean 1960 to 1974)

Temperature (°C)	No. of hours per year at or below given temperature		
	May–October	November–April	Full year
0	11.4	444.0	455.4
–1	5.0	286.9	291.9
–2	1.7	181.5	183.3
–3	0.5	110.7	111.2
–4	0	65.5	65.5
–5	0	34.0	34.0
–6	0	17.7	17.7
–7	0	7.7	7.7
–8	0	4.8	4.8
–9	0	2.0	2.0
–10	0	0.9	0.9

RVO esters in a blend selected as likely to be suitable for year-round use in Ireland.

The fuel economies of the Peugeot during routine work with fuel blends 2 and 3 and mineral diesel were 16.3 km/l (over 5,723 km), 15.9 km/l (over 7,877 km) and 16.9 km/l (over 16,268 km), respectively. Under similar routine driving conditions the Isuzu Trooper achieved 11.0 km/l (over 7,037 km) with fuel blend 1 and 11.2 km/l (over 8,015 km) with mineral diesel. In all cases fuel economy was slightly lower with the biofuels, the difference being from 1.5 to 5.9%. In the road circuit trials, fuel economies with the biodiesel blends were from 1.6 to 4.6% lower than with mineral diesel, and differences between blends were small. None of the differences in the trial reached statistical significance.

The Isuzu Trooper had a significant accumulation of ester in the lubricating oil, reaching about 16% at the end of the trial (Fuel 1, Figure 2). On the other hand, in the Peugeot 360 Xad there was no detectable dilution of the lubricating oils with methyl ester (Fuels 2 and 3, Figure 2). The ester dilution led to a rapid fall in the viscosity of Fuel 1. However, there was no sign of any other adverse effect, such as an increase in viscosity near the end of the oil-change interval. Wear metal levels in

Table 6. Effect of additives on low-temperature properties of two recovered vegetable oil (RVO) ester samples

Sample	Additive	Concentration of additive (mg/kg)	Cloud point (°C)	Cold filter plug point (°C)	Pour point (°C)
1	None		+3	–1	–3
	CP7134	300	+1	–1	–10
	CP7134	600	+2	–4	–12
	Lubrizol	2000	+3	–6	<–18
2	None		+3	–4	–3
	CP7134	300	+1	–1	–5
	CP7134	600	+2	–4	–12
	Lubrizol	2000	+3	–6	–18

Table 7. Low-temperature properties of blends of recovered vegetable oil (RVO) ester and mineral diesel oil

Proportion of RVO ester in blend	Cloud point (°C)	Cold filter plug point (°C)	Pour point (°C)
1.0	+3	–3	–3
0.8	+3	–5	–6
0.6	+3	–8	–6
0.4	+3	–11	–9
0.2	+3	–12	–18
0.0	+3	–15	<–21

the lubricating oils were all within acceptable limits.

The only operational problem of significance to affect the vehicles in the course of the trials arose from inadequate low-temperature properties. Fuel 2, with a CFPP of –3 °C, was in use during the coldest period of the winter when temperatures were occasionally below –10 °C (Figure 3). This led to starting difficulties and fuel filter blockages in the coldest periods.

Discussion

The results of transesterification show that it is possible to produce biodiesel from the RVO that is being collected in Ireland at present. However yields may be low, and high CCR values and variable low-temperature properties are also likely to be a problem. Consequently it may not be possible to use biodiesel derived from RVO on its own. It may need to be blended with biodiesel from other sources or mineral diesel.

A study of Irish temperature data suggests that a fuel for year-round use in the Irish climate should have a CFPP value not higher than –8 °C. To achieve this target with RVO ester, blending with mineral diesel is the most reliable option. Apart from low-temperature problems, the change from mineral diesel to RVO ester as a vehicle fuel may be expected to lead to a slight reduction in fuel economy.

Yields of RVO ester in these trials have been low in comparison with those obtained from refined vegetable oils (Freedman *et al.,* 1984a). This would have a significant effect on the economics of biodiesel production from RVO. Analysis of the glycerol phase has shown that yield losses are due to saponification of the trigyceride, and dissolution of the methyl ester in the glycerol phase by potassium salts of fatty acids.

Since low potassium hydroxide transesterification produced less fatty acids, yield loss due to saponification could be minimised by reducing the amount of potassium

Table 8. Fuel blends used in vehicle performance trials

Full blend	Proportion of methyl ester in blend				Cold filter plug point (°C)
	TME[1]	CME[2]	RVOME[3]	Diesel	
1	0.05	0.30	0.65	0.00	–3
2	0.05	0.00	0.95	0.00	–3
3	0.1	0.00	0.40	0.50	–8

[1]TME = tallow methyl ester.
[2]CME = camelina methyl ester.
[3]RVOME = recovered vegetable oil methyl ester.

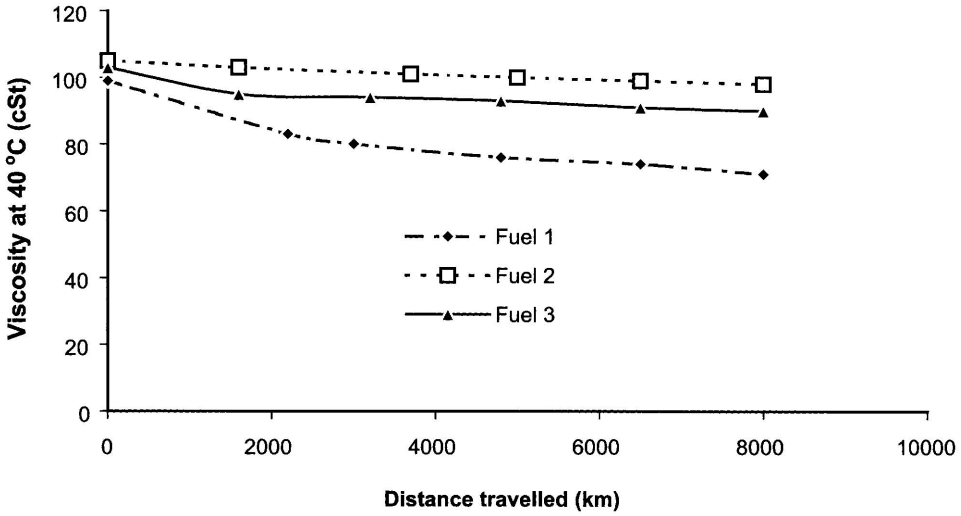

Figure 2: Viscosity variations with three fuels in one oil-charge interval.

hydroxide. However, even if saponification is minimised, free fatty acids in the RVO will end up as potassium salts in the glycerol phase and dissolve some methyl ester. To maximise yields, fatty acids will therefore need to be removed from the RVO or esterified by a different method. Laboratory transesterifications have shown that removal of free fatty acids from the RVO and low potassium hydroxide transesterification (Method 1) gives nearly quantitative yields of methyl ester (Table 3). Considerable yield improvements in the transesterification of animal fats with high free fatty acid concentration have been reported, by acid catalysed conversion of free fatty acids to methyl esters, either before (Canacki and Van Gerpen, 2001) or after (Fröhlich, Rice and Vicente, 2000) base catalysed esterification.

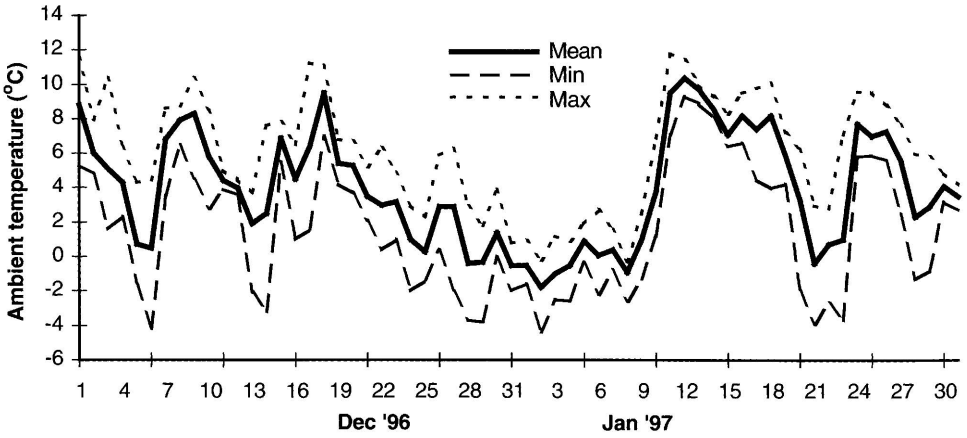

Figure 3: Daily ambient temperatures during coldest period of vehicle performance trials.

References

AOAC (Association of Official Analytical Chemists). 1984. Official Methods of Analysis (14th edition), AOAC, Arlington, VA, USA, 28.028, page 507.

Bailer, H. and de Hueber, K. 1994. 'Handbook of Analytical Methods for Fatty Acid Methyl Esters used as Diesel Fuel Substitutes' (ed. Institute for Chemistry and Technology of Petroleum Products), University of Technology, Vienna, Austria, page 19.

British Standards Institution, BS 2000: Part 15. 1982. Pour point of petroleum oils.

British Standards Institution, BS 2000: Part 219. 1983. Cloud point of petroleum oils.

British Standards Institution, BS 2000: Part 223. 1984. Ash from petroleum products containing mineral matter.

Canakci, M. and Van Gerpen, J. 2001. Biodiesel production from oils and fats with high free fatty acids. *Transactions of the American Society of Agricultural Engineers* **44**(6)**:** 1429–1436.

Christopherson, S.W. and Glass, R.L. 1968. Preparation of milk fat methyl esters by alcoholysis. *Journal of Dairy Science* **52**(8)**:** 1389–1390.

Commission of the European Communities. 2001. Communication from the Commission to the European Parliament, the Council, the Economic and Social Committee and the Committee of the Regions on Alternative Fuels for Road Transportation and on a Set of Measures to Promote the Use of Biofuels. Comm (2001) 547.

Deutsches Institut für Normung, DIN 53 241. 1991. Bestimmung der Iodzahl mit Wijs Lösung.

European Committee for Standardisation. 2001. Automotive fuels – Fatty acid methyl esters (FAME) for diesel engines – Requirements and test methods. Draft prEN 14214. CEN Management Centre, rue de Stassart, B-1050 Brussels.

European Committee for Standardisation, EN 116. 2001. Diesel and domestic heating fuels Determination of cold filter plug point.

Freedman B., Pryde, E.H. and Mounts, T.L. 1984a. Variables affecting the yields of fatty esters from transesterified vegetable oils. *Journal of the American Oil Chemists Society* **61:** 1638–1643.

Freedman, B., Pryde, E.H. and Kwolek, W.F. 1984b. Thin layer chromatography/flame ionization analysis of transesterified vegetable oils. *Journal of the American Oil Chemists Society* **61:** 1215–1220.

Fröhlich, A., Rice, B. and Vicente, G. 2000. The conversion of waste tallow into biodiesel grade methyl ester. *Proceedings of the 1st World Conference on Biomass for Energy and Industry*, Seville, Spain, pages 695–697.

ISO EN 660. 1996. Animal and vegetable fats and oils – determination of acid value and acidity. International Standards Organization, Geneva, Switzerland.

ISO 3104. 1976. Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity. International Standards Organization, Geneva, Switzerland.

ISO 10370. 1993. Petroleum Products – Determination of carbon residue – Micro method. International Standards Organization, Geneva, Switzerland.

ISO 12937. 2000. Petroleum products – Determination of water – Coulometric Karl Fischer titration method. International Standards Organization, Geneva, Switzerland.

Maurer, K. 1991. Pflanzolgewinnung und verwertung. *Landtechnik* **46:** 604–608.

Mittelbach, M. 1996. The high flexibility of small scale biodiesel plants. Production of methyl esters in high quality using various feedstocks. *Proceedings of the 2nd European Motor Biofuels Forum*, Graz, pages 183–187.

Mittelbach, M. 2002. Erfahrungen mit Biodiesel aus gebrauchten Speiseölen in Österreich. *Landbauforschung Volkenrode* **239:** 47–52.

Mittelbach, M. and H. Enzelsberger. 1999. Transesterification of heated rapeseed oil for extending diesel fuel. *Journal of the American Oil Chemists Society* **76:** 545–550.

ÖNORM C1191. 1997. Kraftstoffe - Dieselmotoren, fettsauremethylester, anforderungen. Vienna.

Rice, B., Fröhlich, A. and Leonard, R. 1997. Biodiesel production based on waste cooking oil. ALTENER Contract No. XVII/4.1030/AL/95/IRL, Final Report.

Sams, T., Tieber, J. and Mittelbach, M. 1996. Biodiesel from used frying oil. *Proceedings of the ALTENER Conference on Renewable Energy Entering the 21st Century*, Sitges, pages 1393–1408.

Shriner, R.L., Fuson, R.G. and Curtin, D.Y. 1964. 'Systematic Identification of Organic Compounds', John Wiley & Sons Inc., New York, pages 270–272.

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